TECHNICAL MANUAL

QUALITY CONTROL OF NITROGEN

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INTRODUCTION

1. PURPOSE.

WARNING

Displacement of the air that we breathe by large releases of nitrogen can make oxygen so scarce that asphyxiation and death will result.

This technical manual provides guidance, standards, methods, and procedures for establishing and maintaining quality control over liquid and gaseous nitrogen.

2. SCOPE.

Quality control must be exercised over on-base generation, receipt of nitrogen from a commercial source, transfer, and storage. Quality control procedures of this manual cover general and specific applications as follows:

a. General Applications Such as Purging, Pressurizing, and Cooling – Includes Servicing Aircraft Struts, Accumulators, Tires, and Purging of Aircraft Fuel Controls, and Inerting for Fire Suppression (Chapter 2 and Chapter 5)

b. Pressurizing, Purging, and Blanketing Systems and Support Equipment at Missile Complexes (Chapter 3)

c. Inerting Agent for the C-5 Fire Suppression System (Chapter 2 and Chapter 4)

d. High Purity Inerting Agent (Chapter 6)

CHAPTER 1
GENERAL

1.1 RESPONSIBILITIES.

An effective quality control program requires people from supply, maintenance, engineering, and procurement to work together.

1.2 PERSONNEL.

Personnel selected to perform operations in the nitrogen supply system shall be trained in order to develop a thorough knowledge of the characteristics of nitrogen, its contaminants, and the systems in which it is used. Reliable and knowledgeable personnel are the key to an effective quality control program.

1.3 PHYSICAL AND CHEMICAL PROPERTIES.

Nitrogen may be represented by the chemical formula N_2. It is colorless, odorless, nonflammable, nontoxic, and chemically inactive. Nitrogen may be a gas, a liquid, or a solid depending upon the temperature and pressure. Some physical properties are provided below.

Boiling Point	–320.44°F (–195.8°C) at 14.7 psia
Freezing Point	–345.75°F (–209.86°C) at 14.7 psia
Critical Temperature (Temperature above which nitrogen cannot be liquified by pressure alone.)	–233°F (–147°C)
Critical Pressure (Pressure under which nitrogen may exist as a gas in equilibrium with the liquid at the critical temperature.)	492.3 psia (477.6 psig)
Density at Boiling Point	6.738 pounds per gallon (See Table 1-2 for Nitrogen Weight-Volume Equivalents.)

NOTE

The symbol PSIG represents the expression POUNDS PER SQUARE INCH GAUGE. It is the unit of pressure normally read from a pressure gauge. The sum of the gauge pressure and the barometric pressure is the absolute pressure or true total pressure. Absolute pressure is represented by the term PSIA.

1.4 SOURCE.

Nitrogen is produced from air by air separation plants (generators). Clean, dry, atmospheric air contains approximately 78.1% nitrogen, 20.9% oxygen, and 0.9% argon. The balance consists of carbon dioxide and other trace gases. The process of manufacture includes compression, refrigeration, and expansion cycles to liquify air drawn from the atmosphere. Liquid nitrogen is separated from the liquified air by a distillation process. Water, particulate matter, carbon dioxide, hydrocarbon gases, and other undesirable gases are removed at various stages of manufacture.

1.5 CONTAINERS.

The containers used to transport and store liquid nitrogen are double-walled vessels. The space between the 2 walls has been filled with an insulating material and all the air has been removed to slow the transfer of heat. A pressure-relief valve provides for positive pressure under storage conditions and releases excess nitrogen gas to the atmosphere. Nitrogen gas is formed from liquid nitrogen in proportion to the amount of heat transferred through the container wall. Pressure-relief valves and bursting disks prevent excessive pressure buildup which would burst the container. For specific information and requirements regarding containers and safety-relief devices refer to applicable technical orders, Compressed Gas Association Pamphlets, Parts 100 to 199 of Title 49 of the Code of Federal Regulations (which may be abbreviated 49CFR100-199) and 29CFR1910. Information on gaseous nitrogen containers may be found in the above and in T.O. 42B5-1-2.

1.6 CONTAMINATION.

WARNING

Liquid nitrogen contaminated enough to give the bluish appearance of liquid oxygen requires the same handling procedures as liquid oxygen.

The contaminants (impurities), hydrocarbons (acetylene, gasoline, etc.), moisture, and particulate matter (dust, lint, etc.) in liquid nitrogen must be within use limits for the specific application. Liquid nitrogen is generated at a high enough quality level to allow for a degree of degradation

while being handled, stored, and transported. Liquid nitrogen is a PERISHABLE item; as it ages, it becomes more contaminated. Certain contaminants, to a degree, are present in the liquid nitrogen at time it is delivered from the generator. Contaminants are left behind when liquid nitrogen evaporates; they build up on the inside surfaces of containers and equipment and remain in the liquid. Additional to contaminants existing at time of generation, contaminants are induced at times of handling, storing, and transferring. These contaminants come from such sources as dirty hoses, containers, and the atmosphere. Liquid nitrogen in open containers absorbs oxygen from the air.

The usefulness of liquid nitrogen is prolonged and safety is assured through clean work practices. These characteristics do not apply to gaseous nitrogen. Gaseous nitrogen may be stored indefinitely in high-pressure cylinders and will not degrade. As with liquid, however, contaminants may be introduced during transfer.

1.7 SPECIFICATIONS.

The following specifications apply to the generation and procurement of nitrogen.

a. MIL-PRF-27401, Propellant Pressurizing Agent, Nitrogen, FSC 9135

b. A-A-59503, Nitrogen, Technical, FSC 6830

These specifications contain procurement limits for gaseous (Type I) and liquid (Type II) nitrogen. Procurement limits are listed in Table 1-1. Except for particulate, these limits are the same for both Types I and II. Particulate limits apply to Type II only.

1.8 USE LIMITS.

Use limits establish minimum quality levels related to the end use of the product. There is a second level of quality. Generally, a margin of quality between use limits and procurement limits is necessary to provide for contamination increase during handling and storage. Use limits for applications not specifically covered by this manual may be found in technical manuals of the equipment in which liquid nitrogen is used.

1.9 SAFETY.

WARNING

- Coldness – contact with nitrogen in solid, liquid, or gas form can result in frostbite. Metals and other materials cooled by nitrogen may stick to the skin upon contact and the flesh can become badly burned and torn. Cold nitrogen gas can damage delicate tissues, such as those of the eyes, even when exposure is not long enough to affect the skin of the hands or face. Materials that are soft and pliable at room temperature may easily be broken at the temperatures of liquid nitrogen. Some types of materials and equipment vulnerable to freezing conditions can easily be damaged.

- Expansion – if confined, liquid nitrogen will eventually evaporate and build up a tremendous pressure. The resultant blast could be extremely harmful to both individuals and equipment.

- Asphyxiation – sufficient evaporation of the liquid in a poorly ventilated or unventilated enclosure may cause a dangerous reduction of the oxygen content of the air. Reduced oxygen concentration may produce asphyxiation and death. Because the brain requires the most oxygen of all, a reduction in available oxygen will first affect the mental processes; a slight impairment of the ability to concentrate and think coherently is the first symptom noticed; progression is toward a loss of consciousness. In atmospheres with very high concentrations of nitrogen, unconsciousness can occur in seconds. Recovery in fresh air is rapid and complete, provided that the time of reduced oxygen consumption is not excessively long. Of course, if the reduced oxygen consumption is prolonged, the result is death.

- High-Pressure – this pressure can cause tremendous damage when released uncontrollably. If a valve is broken from a full cylinder it will rapidly accelerate to high speed. A speeding cylinder can penetrate walls and do considerable damage to equipment and injure personnel. Pressurizing systems above their rated pressure can rupture lines and vessels. Ruptured lines will whip and vessel fragments will move at high speed to damage equipment and injure personnel.

Safety for personnel required to work with nitrogen is covered in operation-service manuals and other directives such as AFOSH Standard 91-67.

a. The basic hazards are contact with skin or eyes and displacement of oxygen if large amounts of liquid nitrogen were expelled into a confined area.

b. First Aid.

NOTE

- Do not attempt to rewarm frozen parts until under proper medical care. Control of shock and pain and rewarming of frozen parts must be provided by medical service.
- Remove from exposure immediately. Transport patient to emergency room of hospital/clinic as soon as possible identifying the exposure to liquid nitrogen. Keep dry and warm with blankets en route to emergency room.

1.10 DISPOSITION OF DEGRADED NITROGEN.

When liquid nitrogen has become degraded beyond the use limits, it should be disposed of to the best interests of the government. Liquid nitrogen degraded beyond use limits may sometimes be diverted for use in other equipment able to tolerate the particular degree of degradation; in certain instances, it may be blended with newer material or the only course may be to permit it to gasify into the atmosphere. Occasion may arise when the best interests would be served through condemnation and disposal of liquid nitrogen that is not degraded beyond use limits. As an example, it would not be to the best interests to fill a tank with PERISHABLE propellant which today is within use limits, but which at a later time when about to service a missile will have degraded beyond use limits. Estimating when liquid nitrogen will have degraded beyond the use limits can only be based on past conditions and facts which vary from base to base, and within each base. The WHEN, HOW and POINT of proper disposal of liquid nitrogen, whether or not it is degraded beyond use limits, is a base managerial problem. Specific action to be taken is determined after examination of all conditions and facts.

Table 1-1. Procurement Limits

Specification	MIL-PRF-27401			A-A-59503	
Grade	A	B	C	A	B
Purity (% by volume minimum)	99.5	99.99	99.995	99.95	99.50
Gaseous Impurities (ppm by volume maximum)					
Oxygen	5000	50	20	500	5000
Water	26.3	11.5	5.7	26.3	26.3
Total Hydrocarbons as Methane	58.3	5.0	5.0	50 [1]	50 [1]
Hydrogen	—	—	0.5	—	—
Particulate (mg/liter maximum)	1.0	1.0	1.0	—	—

[1] When specified.

Table 1-2. Nitrogen Weight-Volume Equivalents

Nitrogen						
Mass		Liquid Volume at Boiling Point			Gas Volume at One Atmosphere and 70°F	
Grams	Pounds	Liters	Gallons	Cubic Feet	Liters	Cubic Feet
1.000	0.002205	0.001238	0.0003272	0.00004374	0.8616	0.03043
453.6	1.000	0.5618	0.1484	0.01984	390.8	13.80
807.4	1.780	1.000	0.2642	0.03532	695.7	24.57
3056.0	6.738	3.785	1.000	0.1337	2634.0	93.01
22863.0	50.40	28.32	7.481	1.000	19700.0	695.7
1.161	0.002559	0.001437	0.0003798	0.00005077	1.000	0.03532
32.86	0.07245	0.04070	0.01075	0.001437	28.32	1.000

CHAPTER 2
QUALITY CONTROL OF LIQUID NITROGEN FOR GENERAL APPLICATIONS

2.1 SCOPE.

This chapter applies to activities which produce, store, or use liquid nitrogen for applications other than those identified in Chapter 3, Chapter 5, and Chapter 6.

2.2 ORDERING AND RECEIPT OF LIQUID NITROGEN FROM A CONTRACTOR.

The contractor is required to sample and test the contents of each filled shipping container for compliance with procurement limits (Table 1-1). Government inspection at the contractor facility is not mandatory for all liquid nitrogen applications. Bases that locally procure liquid nitrogen may obtain government inspection, if the need arises, by including the requirement on their purchase order. The inspecting office should be designated on the order and 2 copies of each order should be sent to that office. Upon receipt of product, sampling and testing of the shipping container is not required. Assistance regarding quality problems may be obtained from WR-ALC/AFTT.

2.3 LIQUID NITROGEN SAMPLING AND TEST REQUIREMENTS.

Storage tanks do not need periodic testing. Experience has shown that this grade of product does not degrade significantly during storage. If there is a reason to suspect contamination then storage tanks should be sampled and tested. Unless otherwise specified in this technical order, samples shall be drawn and tested In Accordance With (IAW) Table 2-1 using a sampler conforming to MIL-S-27626 or one that is functionally equivalent. The method of sampling can be found in the sampler technical manual. All samples sent to a laboratory shall be identified by an AFTO Form 176. The applicable blocks of this form shall be completed before attaching it to the sampler.

2.4 TEST LIMITS.

Samples shall be tested for conformance to the procurement limits of A-A-59503, Grade B, including analysis for total hydrocarbons, unless other limits are specified by the submitting activity.

2.5 LABORATORIES.

Samples shall be sent to a laboratory listed in Table 2-2 which is selected in this manner. Find the location of the submitting activity in Table 2-3 and note the number to the right of it. Find this number in Table 2-2 and the designated laboratory for the activity will be listed to the right along with addresses and telephone numbers. For further information or assistance, contact WR-ALC/AFTT, Wright-Patterson AFB, OH 45433-7632, DSN: 785-8050.

Table 2-1. Sampling and Testing Requirements

Source	Test[1]	Conditions	When Sampled	Sampling Location
Air Force Generating Plants	Purity	ALL	According to plant operation manual.	Liquid product line
	Procurement limits[2]	ALL	Once every 45 days and whenever contamination is suspected.	Liquid product line

[1] Samples for purity test shall be analyzed locally. All other samples shall be sent to one of the laboratories as described in Paragraph 2.5 or another laboratory approved by WR-ALC/AFTT for analysis.

[2] When procurement limits of Specification A-A-59503 apply, analysis for total hydrocarbons shall be performed. The limit shall be 50 ppm maximum as methane equivalent.

Table 2-2. Air Force Testing Laboratories

#	Shipping Address	Mailing Address	Telephone Numbers
1	Aerospace Fuels Laboratory DET 3, WR-ALC/AFTLA 2430 C St, Bldg 70, Area B Wright-Patterson AFB, OH 45433-7632	Aerospace Fuels Laboratory DET 3, WR-ALC/AFTLA 2430 C St, Bldg 70, Area B Wright-Patterson AFB, OH 45433-7632	Commercial: (937) 255-2106 DSN: 785-2106
2	Aerospace Fuels Laboratory OL DET 3, WR-ALC/AFTLE 1747 Utah Ave, Bldg 6670 Vandenberg AFB, CA 93437-5220	Aerospace Fuels Laboratory OL DET 3, WR-ALC/AFTLE 1747 Utah Ave, Bldg 6670 Vandenberg AFB, CA 93437-5220	Commercial: (805) 606-6263 DSN: 276-2756
3	Aerospace Fuels Laboratory OL DET 3, WR-ALC/AFTLF - Bldg 725 Unit 5025 RAF Mildenhall, UK APO AE 09459	Aerospace Fuels Laboratory OL DET 3, WR-ALC/AFTLF Unit 5025 APO AE 09459-5025	Commercial: 44-1-638-54-2043 DSN: 314-238-2043/2797/5757
4	Aerospace Fuels Laboratory OL DET 3, WR-ALC/AFTLG - Bldg 854 Unit 5161 Kadena AB, Okinawa, JA APO AP 96368-5162	Aerospace Fuels Laboratory OL DET 3, WR-ALC/AFTLG Unit 5161 APO AP 96368-5162	Commercial: 011-81-611-734-1602/ 3394/0322 DSN: 315-634-3394/1602/3394/0322
5	Aerospace Fuels Laboratory OL DET 3, WR-ALC/AFTLH 15251 Scrub Jay Street, Bldg 54800 Cape Canaveral AFS, FL 32920	Aerospace Fuels Laboratory OL DET 3, WR-ALC/AFTLH 15251 Scrub Jay Street CCAS Patrick AFB, FL 32925-7519	Commercial: (321) 853-5441/5442 DSN: 467-5441

Table 2-3. Laboratory Designations

Location	#	Location	#	Location	#	Location	#
Africa	3	Iceland	1	Missouri	1	Panama	1
Alabama	1	Idaho	2	Montana	2	Pennsylvania	1
Alaska	2	Illinois	1	Nebraska	1	Rhode Island	1
Arizona	2	Indiana	1	Nevada	2	South Carolina	1
Arkansas	1	Iowa	1	Netherlands	3	South Dakota	1
Azores	1	Italy	3	New Hampshire	1	Spain	3
Belgium	3	Kansas	1	New Jersey	1	Tennessee	1
California	2	Kentucky	1	New Mexico	2	Texas	1
Colorado	2	Louisiana	1	New York	1	United Kingdom	3
Connecticut	1	Maine	1	North Carolina	1	Utah	2
Crete	3	Maryland	1	North Dakota	1	Vermont	1
Delaware	1	Mid-East	3	Norway	3	Virginia	1
Florida	5	Massachusetts	1	Ohio	1	Washington	2
Georgia	5	Michigan	1	Oklahoma	1	West Virginia	1
Germany	3	Minnesota	1	Oregon	2	Wisconsin	1
Greece	3	Mississippi	1	Pacific	4	Wyoming	2
Greenland	1						

CHAPTER 3
QUALITY CONTROL OF NITROGEN AT MISSILE COMPLEXES

3.1 SCOPE.

This chapter applies to activities that store or use nitrogen at missile complexes.

3.2 RECEIPT OF LIQUID NITROGEN.

The contractor is required to sample and test the contents of each transport container for compliance with the procurement limits of the current issue of Specification MIL-PRF-27401, Grade A. Government inspection is performed at each contractor shipping point. The delivery of the product is accompanied by a procurement limits test report signed by an authorized representative of the contractor. Upon receipt of product from a contractor, sampling and testing the contents of the transport container are not required before unloading; however, when evidence of possible contamination of the product exists, report the condition to the responsible Government Quality Assurance Representative and to WR-ALC/AFTT before accepting the shipment. Request immediate investigation and appropriate action.

3.3 SAMPLING AND TESTING REQUIREMENTS.

Samples shall be drawn and tested IAW Table 3-2. Test results shall conform to the use limits of Table 3-1.

3.4 CONTAMINATED NITROGEN.

When gaseous or liquid nitrogen is found to exceed the Use Limits (Table 3-1), make proper disposition of the material (see Paragraph 1.10) and check the cleanliness of the equipment in which it was contained. If the equipment does not meet the cleanliness criteria, appropriate cleaning action shall be taken.

3.5 SAMPLING LIQUID NITROGEN.

a. Equipment: Sampler conforming to MIL-S-27626 or one that is functionally equivalent

b. Procedure: According to the Sampler Technical Manual

3.6 SAMPLING GASEOUS NITROGEN.

a. Equipment: A filter/sampler apparatus shall be assembled in the manner diagrammed in Figure 3-1 so that the filter paper is held horizontally and the gas flows down through the filter. The apparatus shall be assembled of the following parts or their equivalent:

1. High-pressure Gas Sampler (Hoke Inc. P/N 8HD1000, NSN 4935-01-087-3946) or (Swagelok P/N 304L-HDF4-1000)
2. Valve, Sampler (Hoke Inc. P/N 2462L84Y) for V3, Figure 3-1
3. Valve with Rupture Disk (Swagelok P/N B-16DKM8-F4-A-2) for V4, Figure 3-1
4. High-pressure Regulator, 7,000 psi Inlet, 2,000 psi Outlet, ¼ inch Aminco Fitting Inlet, ¼ inch AN Fitting Outlet (Circle Seal Controls, Inc. GD720 Series)
5. High-pressure Filter Holder (Millipore P/N XX45-047-00, NSN 6640-00-989-1109)
6. Filter (disc) Type SS, 3-micrometer, 47 mm Diameter (100 discs per package) (Millipore P/N SSWP 047 00, NSN 6640-00-855-8858)
7. O-ring, Teflon-treated Viton, 5 per Package (Millipore P/N XX45 047 05, NSN 5330-00-912-3416)
8. Hose, 72 inches long, Working Pressure 3,000 psi (NSN 4720-00-780-0030)
9. Tubing, Valves, and Connections, as needed. An additional piece of tubing with appropriate connections will be needed to connect C3 with C4 so that the system may be purged with the filter holder removed. This piece of tubing will be referred to as a SPOOL PIECE.

b. Procedure:

(1) Assure that sample cylinder, sample assembly, and flexible hose are clean and in operable condition, and that polyethylene bag enclosing sampler is sealed and unbroken.

(2) Assure that high-pressure filter holder assembly is certified to be clean and prepared for use. Check to assure that the ends are covered with dust caps and that polyethylene bag enclosing the high-pressure filter holder assembly is sealed and unbroken.

(3) Assure that outlets on high-pressure regulator are protected from foreign material with dust plugs or dust caps and are covered with polyethylene bags.

Table 3-1. Use Limits

Total Hydrocarbons as Methane, ppm by Volume	Purity, % by Volume	Water, ppm by Volume	Particulate mg/1 Maximum
200	99.5	26.3 (Dew Point –63.5°F)	0.01 (Gaseous Nitrogen Only)

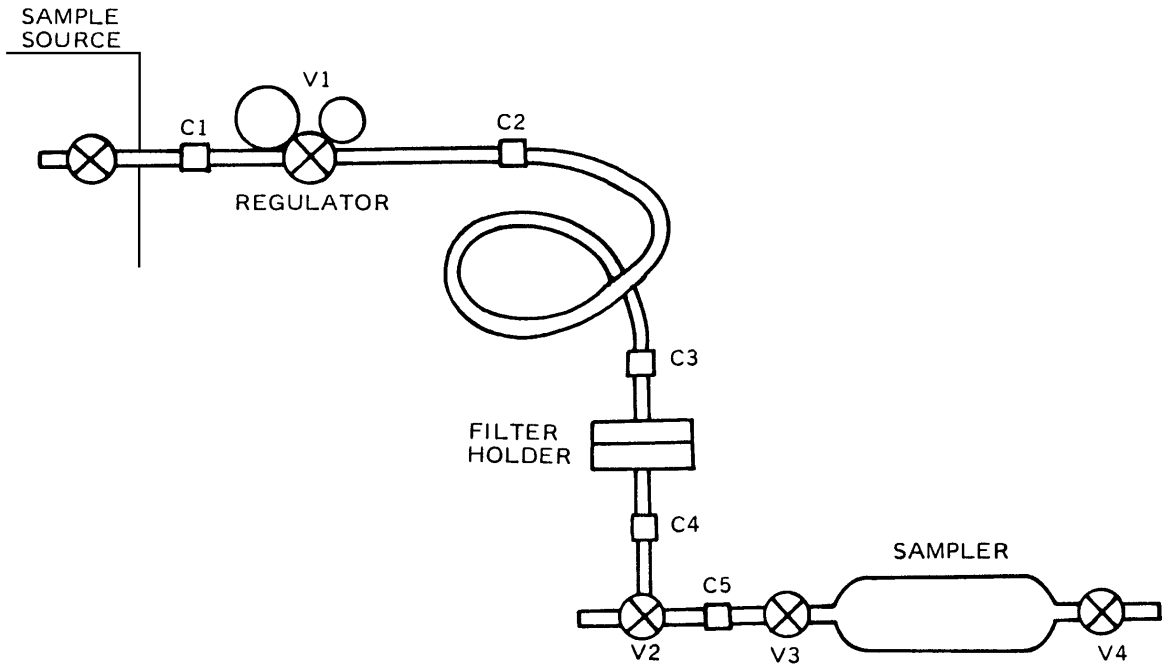

Figure 3-1. Filter/Sampler Apparatus

(4) Momentarily open valve of sample source to clear opening of foreign material.

(5) Attach high-pressure regulator V1 to sample source.

(6) Turn pressure adjusting screw of the regulator counterclockwise until it turns freely.

(7) Connect the apparatus to the sample source using the flexible hose and SPOOL PIECE. The filter holder should not be in the line at this time.

(8) Secure flexible hose to prevent whipping in case of rupture.

(9) Open valve of sample source.

(10) Adjust pressure to 20 psig with regulator.

(11) Purge apparatus for 30 seconds through the bleed valve V2 to clean out foreign material.

(12) Turn pressure adjusting screw of the regulator counterclockwise until it turns freely.

(13) Close bleed valve.

(14) Remove the SPOOL PIECE and connect the filter holder.

(15) Repeat Steps (10) and (11).

Table 3-2. Requirements for Sampling and Testing Air Force Maintained Equipment

Equipment Instructions	Liquid Nitrogen Storage Tanks, Trailers, Rechargers	Gaseous Nitrogen Tube Trailers, Rechargers, Launch Storage Tanks
Frequency	Once every 90 days	Once every 90 days
Sampling Location	Fill/Drain Port	Outlet Fitting [1]
Sampler	MIL-S-27626 or functional equivalent	Filter/Sampler Apparatus See Paragraph 3.6.
Sampling Method	See sampler technical order.	See Paragraph 3.6.
Testing Location	Send samples to laboratory described in Paragraph 3.8.	Particulate test locally. See Paragraph 3.9. Gas – send cylinder to laboratory described in Paragraph 3.8.
Tests	Purity, total hydrocarbons, water	Purity, total hydrocarbons, water, particulate
Quality Control Requirements	Test results shall conform to Table 3-1.	Test results shall conform to Table 3-1.

[1] Equipment described in this chapter is for sampling systems at 6000 psig or less. Outlet fitting selected should not provide a pressure greater than 6000 psig.

(16) Open sampler outlet valve V4 to expel positive pressure and close immediately.

(17) Open sampler inlet valve V3 and slowly, in approximately 2 minutes, increase the regulated pressure to 1800 ± 20 psig.

(18) Record the final pressure of the sampler on the Sample Identification and Control Tag.

(19) Close sampler inlet valve V3.

(20) Close valve of sample source.

NOTE

Apparatus must be depressurized on the sampler side of the filter holder to prevent reverse flow from blowing particulates off the filter.

(21) Bleed residual pressure through bleed valve V2.

(22) Immediately after disconnecting, cap both ends of the filter holder and place in a polyethylene bag.

(23) Seal the polyethylene bag.

(24) Complete 2 AFTO Forms 176 as follows:

(a) Enter type of nitrogen in ITEM block.

(b) Enter MIL-PRF-27401 in SPECIFICATION block.

(c) Enter all information identified in SUBMITTED BY block.

(d) Enter base sample number.

(e) Enter source of sample in the NOTES block.

(f) Enter pressure of sampler as submitted.

(g) Enter date sampled.

(25) Attach 1 tag to the polyethylene bag.

(26) Place dust caps on sampler valves.

(27) Attach second tag to the sampler.

(28) Take filter holder to base laboratory for weighing of filter paper. Keep filter holder in an upright position until filter paper has been removed.

(29) Package and ship sampler to the laboratory designated in Paragraph 3.8.

3.7 SAMPLING AND TESTING RECORDS.

Base personnel shall maintain a complete and accurate log on the quality control of nitrogen, listing information and data as follows:

a. Equipment (Base Liquid Nitrogen Trailer, Base Gaseous Nitrogen Trailer, Recharger — Liquid or Gaseous Nitrogen) and Identification Number

b. Sample Identification and Control Number

c. Laboratory Report and Control Number

d. Test Results and Date Reported

e. Actions Taken as Required by Test Results

3.8 DESIGNATED LABORATORY.

Testing for purity, moisture, and hydrocarbons shall be performed by the Aerospace Fuels Laboratory at Vandenberg AFB or Cape Canaveral AFS, FL. For testing at other locations, contact WR-ALC/AFTT, Wright-Patterson AFB, OH 45433-7632, DSN: 785-8050 for approval.

a. Address – for shipping addresses to Vandenberg AFB and Cape Canaveral AFS refer to Table 2-2.

b. Test Methods – samples of nitrogen shall be tested IAW the test methods of the current issue of Specification MIL-PRF-27401, except for purity. Purity may be determined by either the methods of MIL-PRF-27401 or by a volumetric gas analysis apparatus (Orsat type) using a suitable oxygen-absorbing reagent.

3.9 BASE LABORATORY.

Gaseous nitrogen shall be tested locally for particulate. This testing shall consist of initially weighing the filter paper, assembling the filter holder, sampling the nitrogen (Paragraph 3.6), finally weighing the filter paper, and calculation of the result.

a. Initial Weighing of the Filter Paper – a new Millipore, Type SS, 3 micron filter paper shall be dried and weighed prior to cleaning the high-pressure filter holder assembly so that it can be immediately placed in the filter holder as it is assembled after cleaning. The procedure is as follows:

(1) Using forceps, remove a filter paper from its container and place in an open petri dish. Mark the cover of the petri dish to suitably identify the petri dish and the filter paper, stored therein, for later reference.

(2) Place petri dish (with filter paper) for 1 hour in a vacuum oven held at 70°C (158°F). Leave cover off petri dish to allow for proper drying of filter paper.

(3) Remove petri dish from the vacuum oven and place it (with filter paper) for 30 minutes in a desiccator, containing desiccant.

(4) Using forceps, remove the filter paper from the petri dish and place it in position on the balance. Wait 2 minutes to allow filter paper to come to constant weight. Record the constant weight to within 0.1 mg or better.

(5) Using forceps, remove the filter paper from the balance and place it in the petri dish (within the desiccator). Place cover on petri dish, and close the desiccator. Store the filter paper in desiccator until it is to be installed in the cleaned high-pressure filter assembly.

b. Final Weighing of the Filter Paper.

(1) Keep the high-pressure filter assembly in an upright position.

(2) Disassemble the unit and using forceps, carefully remove the filter paper from the top of the sintered back-up disc.

(3) Place the filter paper in a petri dish.

(4) Place petri dish (with filter paper) for 30 minutes in a vacuum oven held at 70°C (158°F). Leave cover off petri dish to allow for proper drying of filter paper.

(5) Remove petri dish with filter paper from the vacuum oven and place it in a desiccator, containing desiccant, for 30 minutes.

(6) Using forceps, remove the filter paper from the petri dish and place it in position on the balance. Wait 2 minutes to allow filter paper to come to constant weight. Record the constant weight to within 0.1 mg, or better.

c. Calculation of the Particulate Content.

(1) Determine the net weight of particulate by subtracting the initial weight of the filter paper from its final weight.

NOTE

The calculation below is based on a sampler which has a water volume of 1000 ml and is pressurized to 1800 ± 20 psig. One thousand milliliters or 1 liter of gas at 1800 psig is equal to 123 liters at atmospheric pressure.

(2) Calculation.

$$\text{Particulate (mg/1)} = \frac{\text{net weight (mg)}}{\text{123 liters}}$$

3.10 PREPARATION OF SAMPLING EQUIPMENT.

3.10.1 Cleaning Equipment and Materials.

a. Filter Paper (Membrane), 0.45 micrometer, 47 mm in Diameter (Millipore P/N HAWP 047 00, NSN 6640-00-083-5308 or equal)

b. Filter Holder, Hydrosol (Millipore P/N XX20 047 20, NSN 6640-00-893-3096 or equal)

c. Distilled Water

d. Laboratory Glassware Detergent, Liqui-Nox (NSN 7930-01-189-3977) or equivalent

3.10.2 Filtration of Distilled Water. Prior to cleaning the sampling equipment, the distilled water shall be filtered IAW the following procedures:

a. Wash base and funnel components of the hydrosol filter holder and a laboratory filter flask with detergent and water. Thoroughly rinse with hot, soft tap water.

b. Assemble the filter holder with a 0.45 micron filter paper in place.

c. Filter the desired volume of distilled water through the filter into a clean, dry filter flask. Remove entire filter assembly and transfer filtrate to a clean, dry stock bottle.

3.10.3 Cleaning Procedures for High-Pressure Filter Holder. The high-pressure filter assembly and closure caps shall be cleaned before each use IAW the following procedures:

a. Wash thoroughly in a solution of detergent and hot water. Rinse twice in hot tap water.

b. Rinse twice with filtered distilled water.

c. After rinsing, dry all components in an oven at 120° – 130°F.

d. Partially assemble the components of the filter holder.

e. Place the filter paper (previously dried and weighed, and its weight recorded, IAW Paragraph 3.9) on the sintered back-up disc and complete the assembly of the high-pressure filter holder.

f. Cap off both ends of the filter holder.

g. Place the filter holder in polyethylene bag and seal bag.

h. Prepare and sign a tag certifying that the filter holder has been cleaned IAW this chapter.

i. Attach tag to filter holder.

3.10.4 Cleaning Procedure for Flexible Hoses. The flexible hoses shall be cleaned before each use as follows:

a. Wash the flexible hoses inside and outside with a solution of detergent and hot water. Rinse twice with hot tap water.

b. Rinse twice with filtered distilled water and place in a vertical position to drain.

c. Dry the flexible hoses in an oven at 120° – 130°F.

d. Protect inside of flexible hose from foreign material by taping polyethylene bags on both ends.

CHAPTER 4
QUALITY CONTROL OF LIQUID NITROGEN FOR C-5A FIRE SUPPRESSION SYSTEM

4.1 NOTICE.

The requirements of this chapter have been deleted. Experience has shown that this grade of product does not degrade significantly during storage. Refer to Chapter 2 for general requirements.

CHAPTER 5
QUALITY CONTROL OF GASEOUS NITROGEN FOR GENERAL APPLICATIONS

5.1 SCOPE.

This chapter applies to activities which produce, store, or use gaseous nitrogen for applications other than those identified in special chapters.

5.2 ORDERING AND RECEIPT OF GASEOUS NITROGEN FROM A CONTRACTOR.

The contractor is required to sample and test the gaseous nitrogen for conformance to the applicable specification (Paragraph 1.7). Government inspection at the contractor facility is not mandatory for all gaseous nitrogen applications. Bases that locally procure liquid nitrogen may obtain government inspection, if the need arises, by including the requirement on their purchase order. The inspecting office should be designated on the order and 2 copies of each order should be sent to that office. Sampling and testing of gaseous nitrogen is not required upon receipt. Assistance regarding quality problems may be obtained from WR-ALC/AFTT.

5.3 SAMPLING AND TESTING REQUIREMENTS.

When samples are to be sent to a laboratory they shall be taken and identified according to Paragraph 5.4 and Paragraph 5.5. Approved laboratories are listed in Table 2-2. Other laboratories may be approved by WR-ALC/AFTT. Samples shall be tested for conformance to the use limits of Paragraph 5.6.

a. Liquid air separation plants.

(1) Samples shall be drawn as required by the plant operation technical order. These samples shall be tested locally.

(2) Samples shall be taken at 45-day intervals whenever production run schedules permit and sent to a laboratory as described above. If a plant is not in production at the end of a 45-day interval then a sample shall be taken during the next production run. Other samples may be taken as appropriate. For example, samples should be taken when contamination is suspected.

b. Self Generating Nitrogen Servicing Cart – samples shall be taken and tested as required by the cart operation technical order. These samples shall be tested locally. If additional testing is needed to solve a quality problem, samples may be sent to a laboratory as described above.

c. Base Storage Cylinders and Tubes – periodic testing of base storage cylinders and tubes is not necessary. If testing is needed to solve a quality problem, samples may be sent to a laboratory as described above.

5.4 GASEOUS SAMPLER AND SAMPLING METHOD.

WARNING

A sampler must not be filled to a pressure greater than its rated pressure. See the discussion on high-pressure in Paragraph 1.9.

When a sample is to be sent to a laboratory, use the sampler and method described below.

The gaseous sampler shall consist of the cylinder section from the cryogenic sampler as follows:

a. Cylinder Assembly – NSN 3655-01-059-8222YD

b. Adapter – NSN 4730-01-128-8257YD

c. Seal – NSN 5330-01-129-0621

Refer to T.O. 33D2-10-60-1 for assembly and purging procedures. The sample shall be taken from the charging manifold using the method of ASTM F 307 except that Step 6.7 is deleted.

5.5 SAMPLE IDENTIFICATION.

When a sample is to be sent to a laboratory, complete the applicable blocks of an AFTO Form 176. In the block labeled ITEM enter NITROGEN, T.O. 42B7-3-1-1, 5-6a or NITROGEN, T.O. 42B7-3-1-1, 5-6b in order to show which limits apply. Attach the form to the sampler.

5.6 USE LIMITS.

a. Servicing aircraft tires, struts, and accumulators.

Purity	– 95% by volume, minimum
Oxygen	– 5% by volume, maximum
Water Vapor	– 26 ppm by volume, maximum

Samples shall be tested using the methods of A-A-59503.

b. Other Applications – samples shall be tested for conformance to the procurement limits of A-A-59503, Grade B, including analysis for total hydrocarbons, unless other limits are specified by the submitting activity.

5.7 USE, HANDLING, AND MAINTENANCE OF COMPRESSED GAS CYLINDERS.

Refer to T.O. 42B5-1-2. Maintenance performed on Air Force cylinders by contractors shall be according to MIL-STD-1411.

CHAPTER 6
QUALITY CONTROL OF HIGH PURITY LIQUID NITROGEN

6.1 SCOPE.

This chapter applies to those activities who store liquid nitrogen used as a high purity inerting agent.

6.2 PROCUREMENT LIMITS.

Liquid nitrogen procured from a commercial source or generated by the Air Force must meet the requirements of the current issue of Military Specification MIL-PRF-27401, Type II, Grade B. This specification may be modified as described below when the nitrogen is to be used in hydrocarbon fuels systems.

a. The test requirement for total hydrocarbons is deleted. The value of testing for gaseous hydrocarbons is low since nitrogen is used in a system already loaded with hydrocarbon fuels.

b. The purity value is determined by summing the percentages for oxygen and water content and subtracting the sum from 100%.

6.3 ORDERING AND RECEIPT OF LIQUID NITROGEN FROM A CONTRACTOR.

The contractor is required to sample and test the contents of each filled shipping container for compliance with procurement limits (Table 1-1). Government inspection is recommended. The requirement for inspection should be included on the contract and/or purchase order. The inspecting office should be designated on the order and two copies of each order should be sent to that office. Upon receipt of product, sampling and testing of the shipping container is not required. Assistance regarding quality problems may be obtained from WR-ALC/AFTT.

6.4 LIQUID NITROGEN SAMPLING AND TEST REQUIREMENTS.

Samples shall be drawn and tested according to Table 6-1 using a sampler conforming to MIL-S-27626 or one that is functionally equivalent. The method of sampling can be found in the sampler technical manual. All samples shall be identified by an AFTO Form 176. The applicable blocks of this form shall be completed before attaching it to the sampler.

6.5 USE LIMITS.

Samples shall be tested for conformance to the procurement limits of A-A-59503, Type II, Grade A, including analysis for hydrocarbons. There is no use limit for hydrocarbons when product is used for inerting hydrocarbon fuels systems and values will be reported for information only.

6.6 TEST REPORTS.

A test report shall be prepared for each sample. One copy shall be retained, one shall be forwarded to the submitting activity, and one shall be forwarded to WR-ALC/AFTT.

6.7 LABORATORIES.

The following laboratories or other laboratories authorized by WR-ALC/AFTT may be used to test high purity nitrogen:

a. Aerospace Fuels Laboratory, RAF Mildenhall, England

b. Aerospace Fuels Laboratory, Kadena AB, JA

c. Aerospace Fuels Laboratory, Wright-Patterson AFB, OH

d. Aerospace Fuels Laboratory, Vandenberg AFB, CA

e. Aerospace Fuels Laboratory, Cape Canaveral AFS, FL

See Chapter 2 for complete addresses.

Table 6-1. Sampling and Testing Requirements

Source	Test	Conditions	When Sampled	Sampling Location
Air Force Generating Plants	Purity [1]	ALL	According to plant operation manual.	Liquid product line
	Purity and hydrocarbons [1]	ALL	Once every 45 days and whenever contamination is suspected.	Liquid product line
Air Force Generating Plant Storage Tanks	Purity [2]	1. Continuous production. 2. Intermittent production.	1. Once each week. 2. After each start-up.	Drain valve
	Purity and hydrocarbons [2]	ALL	Once every 90 days.	Drain valve
Air Force Storage Tanks (100-gallon capacity or more)	Purity and hydrocarbons [2]	ALL	Once every 90 days.	Any port from which liquid nitrogen can be drawn.

[1] Samples shall be tested for conformance to the procurement limits – Specification MIL-PRF-27401, Grade B.
[2] Samples shall be tested for conformance to the use limits – Specification A-A-59503, Grade A.